

## P. Mechanical Behavior of Ceramic Materials for Heavy-Duty Diesel Engines

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### Objectives

- Enable the development of more wear-resistant and mechanically robust ceramic components through the systematic evaluation of contact damage in ceramics.
- Characterize rate effects on deformation processes in  $\text{Si}_3\text{N}_4$ .
- Develop supplemental models to assist in the interpretation of deformation.

### Approach

- Characterize damage processes in  $\text{Si}_3\text{N}_4$  ceramics through instrumented static and dynamic indentation and instrumented scratch testing.
- Develop criterion that can be used to rank resistance to damage.
- Evaluate relevant  $\text{Si}_3\text{N}_4$  grades (e.g., existing commercial roller element and ball bearing compositions, standard reference materials).
- Evaluate grain size effects on damage processes through static and dynamic indentation (model  $\text{Al}_2\text{O}_3$  ceramics used).

### Accomplishments

- Established dynamic indentation and instrumented scratch testing facilities.
- Developed a new brittleness parameter that represents static and dynamic indentation responses.
- Developed a generic finite element method (FEM) that predicts the onsets of quasi-plasticity and fracture/cracking in any target material as a function of indentation load, indenter material, friction, and confinement.
- Co-developed and copyrighted  $\mu$ -FEA software.

### Future Direction

- Conduct a complete study of grain size effects on rate and deformation processes.
  - Begin a new project involving reliability analysis and design optimization of piezostack fuel injectors for diesel engines.
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## Introduction

The application of more ceramic components in engines and transportation systems would be enabled if they could be confidently used in more wear-related applications and if they could be manufactured and machined faster (i.e., more cost-effectively). This would occur if contact-induced damage mechanisms (that limit their mechanical performance or dictate their machinability) were understood, predictable, and controlled.

This project quantifies the loads and stresses associated with the initiation of “quasi-plasticity” and fracture processes in ceramics using instrumented indentation and scratch testings. Understanding their competition and interaction is a critical part of this project as well. Traditional structural ceramics (e.g.,  $\text{Si}_3\text{N}_4$ ) under consideration for diesel engine components are of primary focus.

Through the quantification of the loads or stresses that initiate quasi-plasticity and fracture, contact-induced damage mechanisms in ceramics are characterized and modeled and linked to micro-structure, and ultimately to wear performance, the optimization of machining, and rolling contact fatigue. Additionally, the influence that independent parameters such as grain size, loading rate, confinement or residual stresses, temperature, field effects have on the evolution of contact-induced damage are also examined. The thorough understanding of the competition of fracture and quasi-plasticity in ceramics will enable improved and faster means of ceramic component manufacturing and surface engineering (e.g., ductile regime machining) and maximize mechanical performance when surface condition (e.g., bending) or when surface-located events (e.g., wear, impact) are service-life-limiters in engine and transportation system components.

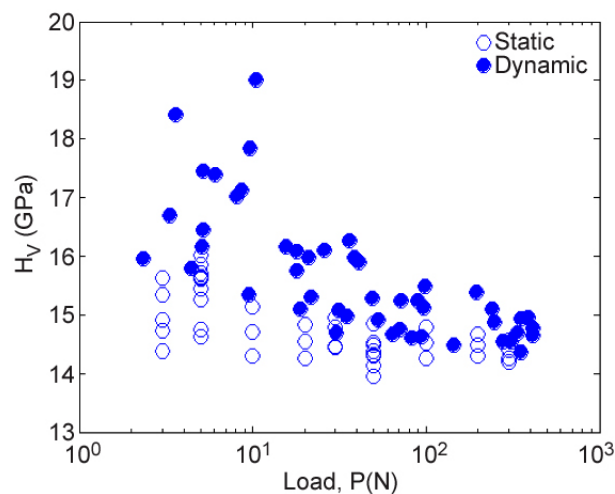
## Approach

There were four primary components to the FY 2005 work. Instrumented indentation and static indentation and instrumented scratch testing were used to characterize contact-induced damage processes in  $\text{Si}_3\text{N}_4$ . A criterion was developed that can be used to rank resistance to contact-induced damage. Several grades of  $\text{Si}_3\text{N}_4$  were evaluated (e.g., existing roller element and ball bearing compositions as well as standard reference materials). Lastly, grain size effects (in model  $\text{Al}_2\text{O}_3$

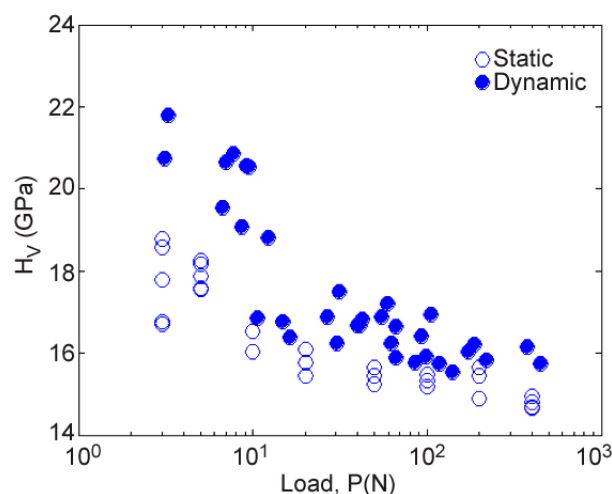
ceramics) on damage processes from indentation and scratch testings were examined.

## Results

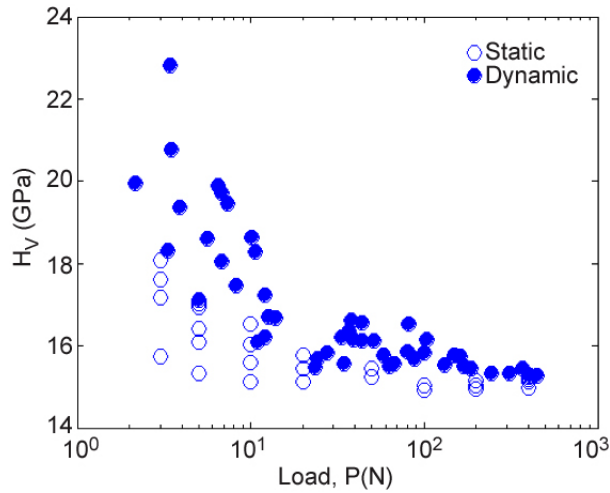
Static and dynamic indentation hardnesses of Ceralloy 147-31N, NBD200, SN101C, TSN-03NH, and NC132 silicon nitrides were measured as a function of load and their results are respectively shown in Figures 1–5. The rationale for testing each grade: the 147-31N grade is presently used as a cam



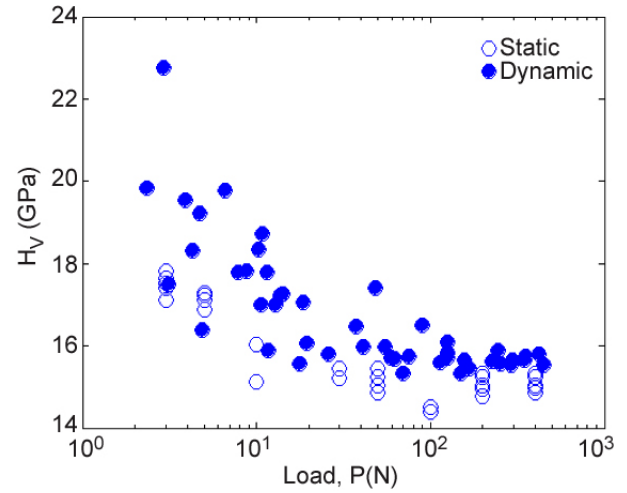
**Figure 1.** Vickers Hardness for Ceralloy 147-31N  $\text{Si}_3\text{N}_4$  as a function of load for quasi-static and dynamic indentation.



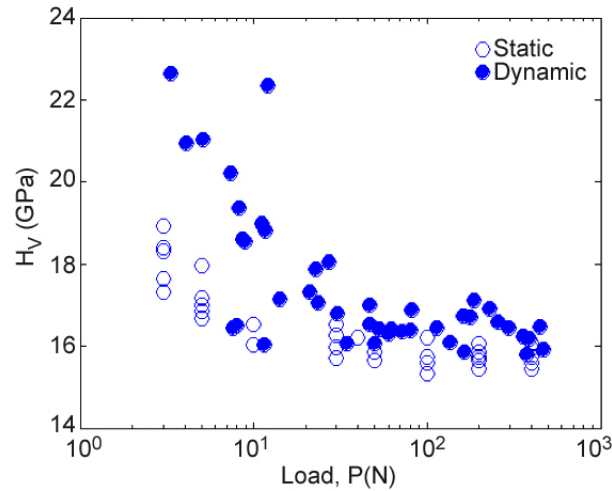
**Figure 2.** Vickers Hardness for NBD200  $\text{Si}_3\text{N}_4$  as a function of load for quasi-static and dynamic indentation.



**Figure 3.** Vickers Hardness for SN101C  $\text{Si}_3\text{N}_4$  as a function of load for quasi-static and dynamic indentation.



**Figure 4.** Vickers Hardness for TSN-03NH  $\text{Si}_3\text{N}_4$  as a function of load for quasi-static and dynamic indentation.



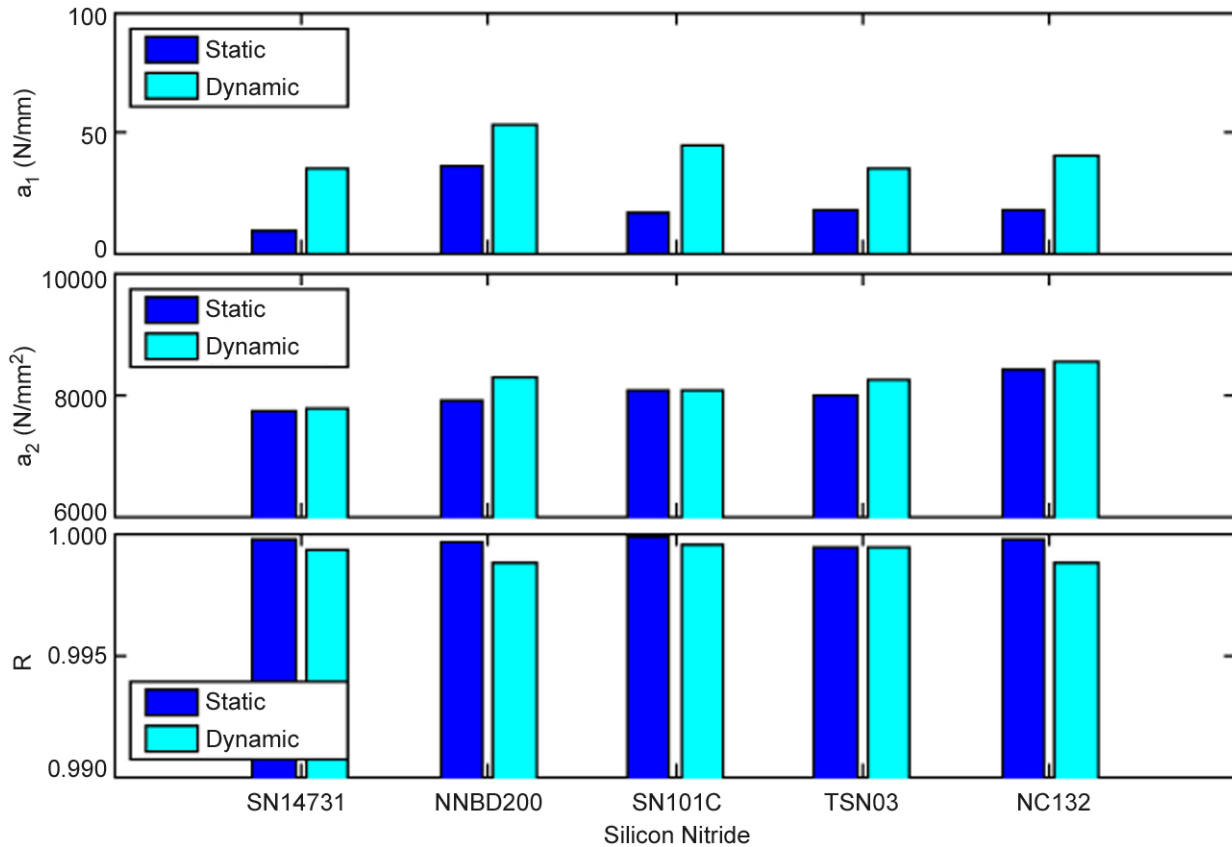
**Figure 5.** Vickers Hardness for NC132  $\text{Si}_3\text{N}_4$  as a function of load for quasi-static and dynamic indentation.

roller follower material, so interest exists to quantify its indentation and scratch responses, NBD200, SN101C, and TSN-03NH are ball bearing grades so their testing is relevant to their examination of rolling contact fatigue behavior, NBD200 is a NIST SRM for Knoop hardness, and NC132 is a NIST SRM for fracture toughness so they serve as references upon performance comparison among all the silicon nitrides. An indentation size effect (ISE) and a rate effect are exhibited by all (increased  $H$  and greater ISE with rate).

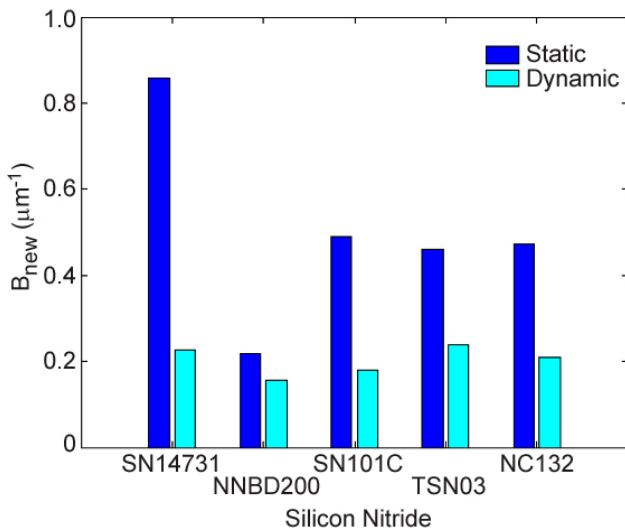
A good fit to the experimental data is often obtained using a formula that Frohlich et al.,<sup>1</sup> and Li and Bradt<sup>2</sup> attribute to Bernhardt<sup>3</sup>

$$P = a_1 d + a_2 d^2, \quad (1)$$

where  $P$  is indentation load,  $d$  is indent diagonal size, and  $a_1$  and  $a_2$  are constants. The  $a_1$  term is relatable to the energy consumed in creating new surfaces,<sup>1-2</sup> whereas the  $a_2$  term is relatable to the volume energy of deformation.<sup>4</sup> The determination of those terms for each of the silicon nitrides as a function of rate is shown in Figure 6. Additionally, a new brittleness parameter was developed that involves the use of the ratio of  $a_2/a_1$  because it can provide insight into the competition of those two mechanisms. The brittleness of the five silicon nitrides is shown in Figure 7, and the relation shows



**Figure 6.** Comparison of  $a_1$  and  $a_2$  terms [see Eq. (1)] for the five silicon nitrides when indentation was performed statically and dynamically.

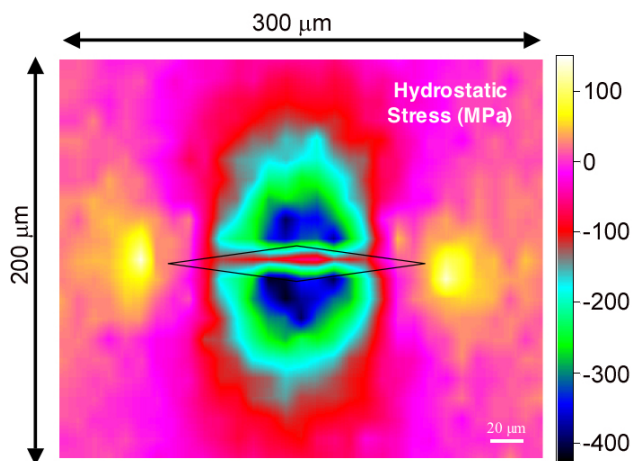


**Figure 7.** Comparison of a new Brittleness parameter ( $a_2/a_1$ ) for the five evaluated silicon nitrides for static and dynamic loadings. The trends show that all the grades act less brittle when they were dynamically loaded.

that all five silicon nitrides respond in a less brittle fashion when they were dynamically indented.

The exploration of the effect of indentation on residual stress generation was initiated using optical fluorescence. An illustration of this is shown in Figure 8 for a 99.9% alumina that was Knoop indented at 2 kg. A maximum tensile stress of  $\sim 100$  MPa is observed at the tip of the cracks that come from the Knoop indent diagonals. Quantification of these effects will enable engineering control of ceramic machining or allowable wear conditions to inhibit or avoid deleterious effects that may be more strongly linked to fracture or quasi-plastic processes or both.

An ANSYS Hertzian indentation model that was developed in FY 2004 continued to be refined in FY 2005. A three-dimensional (3-D) model was developed in FY 2005 using “concrete” elements that will enable the prescription of strength asymmetry in tension and compression. A tensile strength value will be imputable that will be associated with fracture processes, whereas a

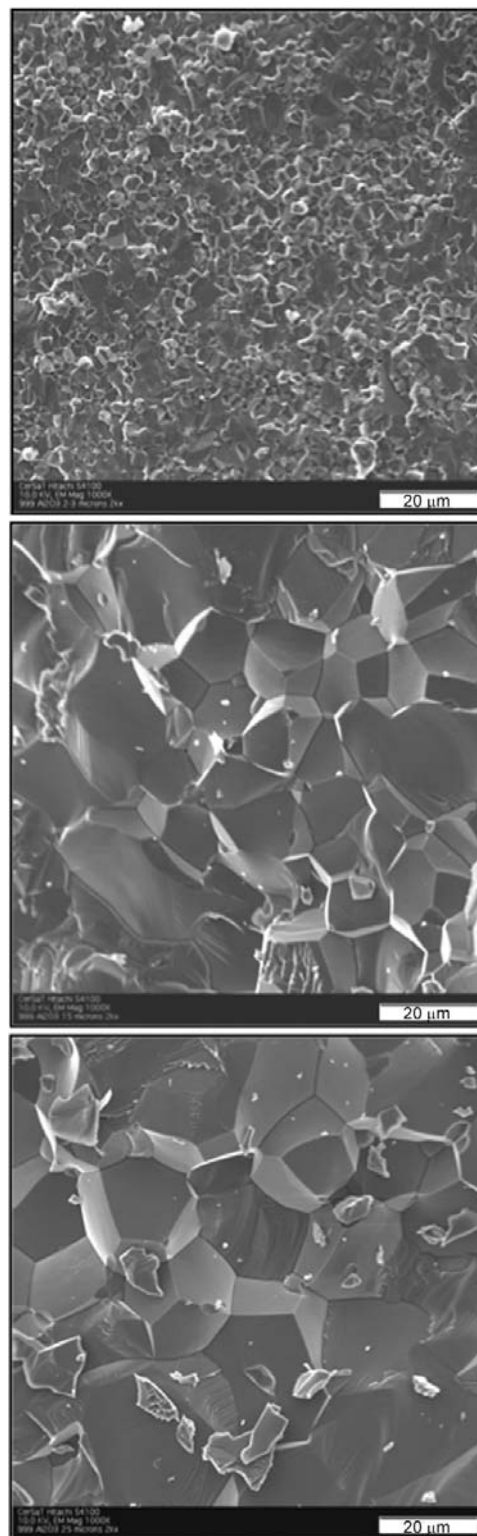


**Figure 8.** Hydrostatic stress map generated with optical fluorescence about a 2-kg Knoop indent in a 99.9% alumina having a ~2- to 3- $\mu$ m average grain size.

compressive strength value will be imputable that will be associated with contact-induced quasi-plastic deformation mechanisms.

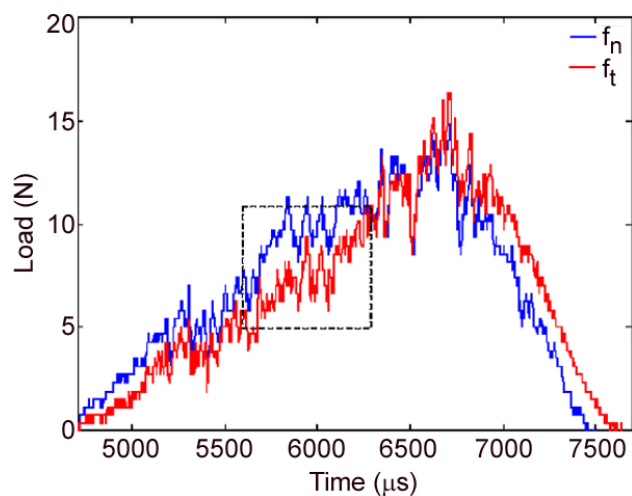
A substantial amount of instrumented scratch characterization occurred during the latter half of FY 2005. Testing of silicon nitride focused on the evaluation of Ceradyne's Ceralloy 147-31N, which is a reaction-bonded material used in cam roller followers. Evaluation of three 99.9% aluminas having mean sizes of 2, 15, and 25  $\mu$ m (see Figure 9) also proceeded; the rationale behind the alumina testing is to evaluate scratch response as a function of controlled grain size with the same chemical composition. Additionally, the testing of alumina affords the opportunity to map out residual stresses about the contact or scratch damage using optical fluorescence. Scratch damage evolution and its quantification are being related to machining and wear performance.

The interpretation of the load histories during scratch testing with respect to microstructural examination is ongoing. Normal and tangential forces are continuously monitored during the (pendulum) scratch test. Examples of these with associated microstructural damage are shown in Figures 10–11 for Ceralloy 147-31N, and Figures 12–17, for the 2-, 15-, and 25- $\mu$ m alumina, respectively. The effects of lateral crack formation on normal and tangential load histories and depth changes are illustrated in Figure 18. SEM observations show that there is an apparent grain size effect on the scratch responses; in particular,

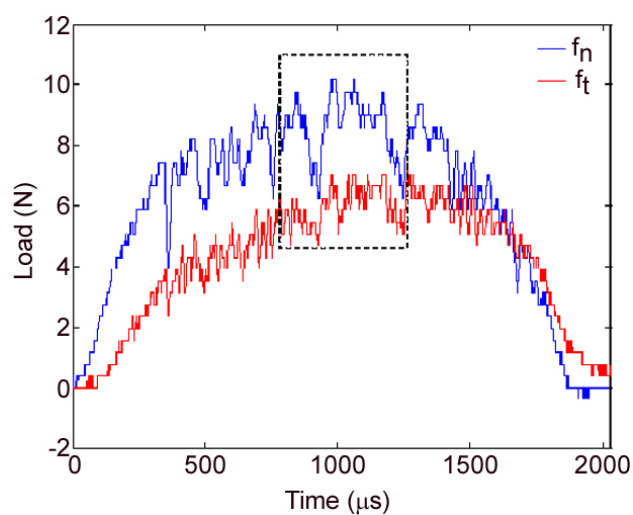


**Figure 9.** Microstructure of 99.9% aluminas with mean average grain size of (top) 2  $\mu$ m, (middle) 15  $\mu$ m, and (bottom) 25  $\mu$ m.

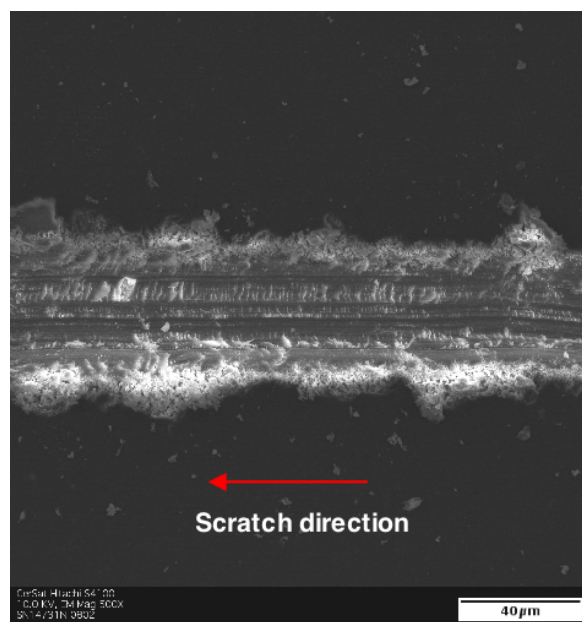




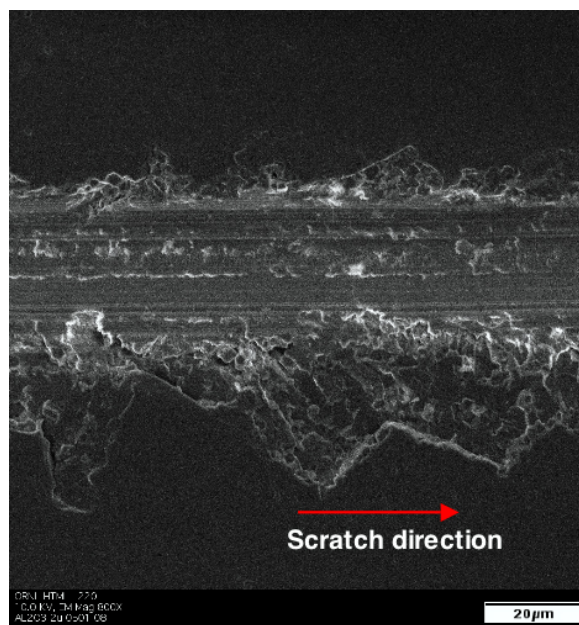
**Figure 10.** Example of normal (blue) and tangential (red) load histories during a Ceralloy 147-31N silicon nitride scratch test.



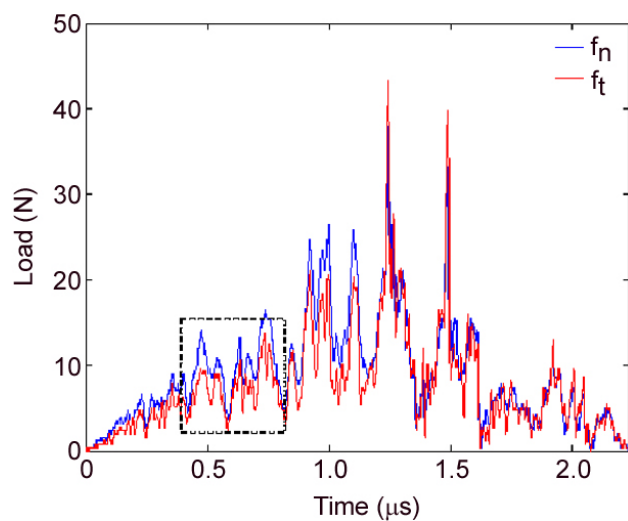
**Figure 12.** Example of normal (blue) and tangential (red) load histories during a 99.9% 2-μm alumina scratch test.



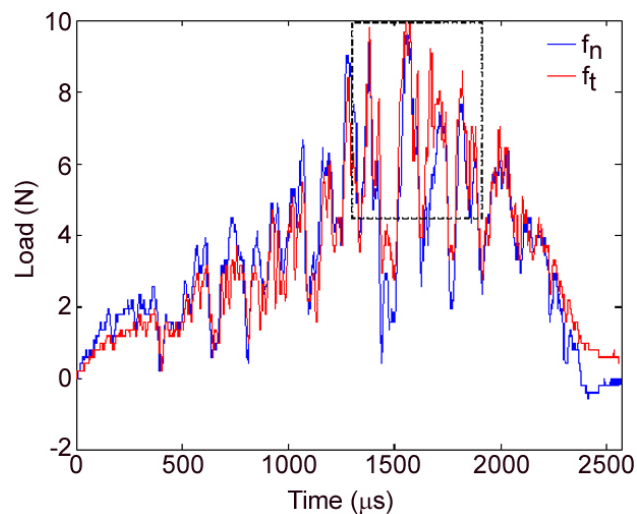
**Figure 11.** Scratch on Ceralloy 147-31N associated with boxed region in Figure 10.



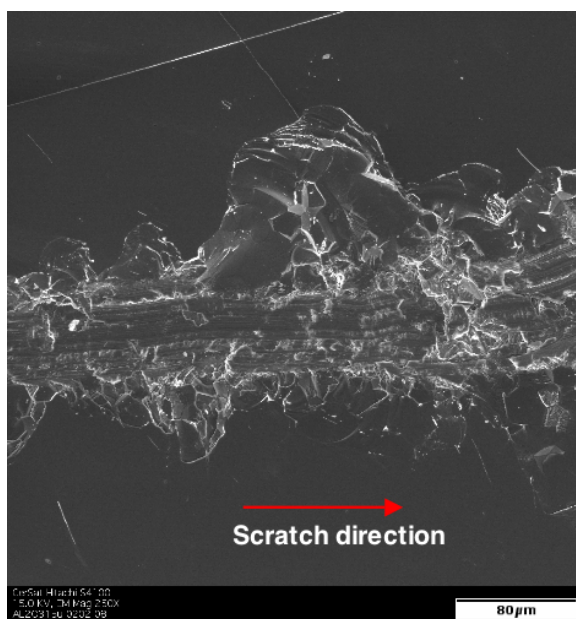
**Figure 13.** Scratch on 99.9% 2-μm alumina associated with boxed region in Figure 12.



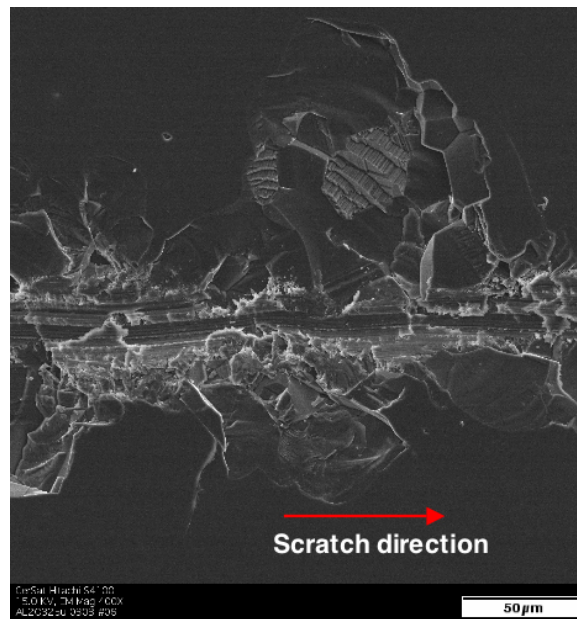
**Figure 14.** Example of normal (blue) and tangential (red) load histories during a 99.9% 15- $\mu\text{m}$  alumina scratch test.



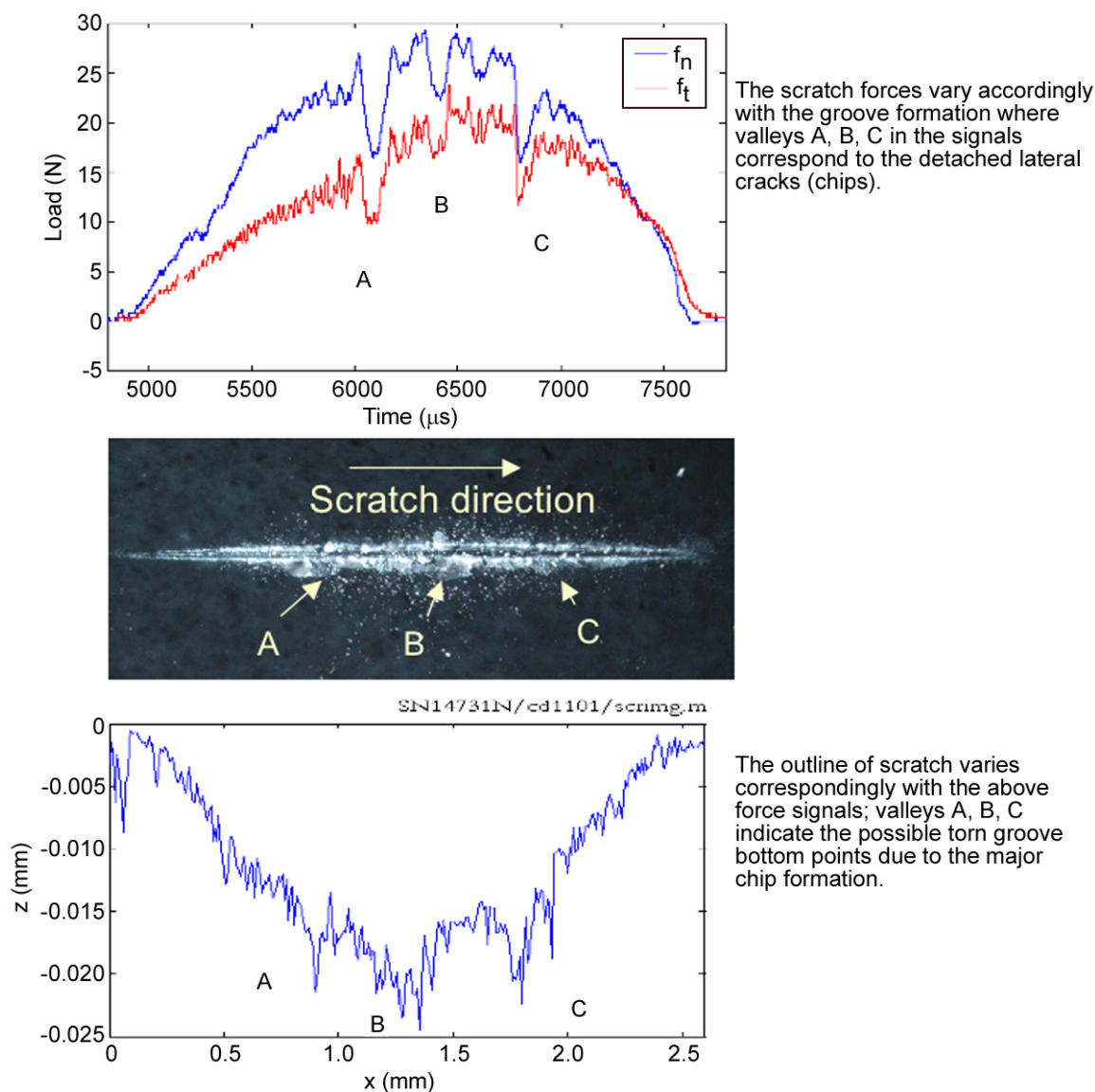
**Figure 16.** Example of normal (blue) and tangential (red) load histories during a 99.9% 25- $\mu\text{m}$  alumina scratch test.



**Figure 15.** Scratch on 99.9% 15- $\mu\text{m}$  alumina associated with boxed region in Figure 14.



**Figure 17.** Scratch on 99.9% 25- $\mu\text{m}$  alumina associated with boxed region in Figure 16.



**Figure 18.** Evidence of lateral crack formation evident in the normal and tangential load histories (top graph) and in depth changes too (profilometry—bottom graph).

scratch forces oscillate more significantly during the scratching of larger-grained alumina corresponding to a greater amount of lateral crack formation.

$\mu$ -FEA, a “microstructure-based finite-element analysis” computer program, was copyrighted in FY 2005.  $\mu$ -FEA is a graphics-user-interfaced LabVIEW executable program that serves as a pre- and post-processor to the ANSYS finite-element analysis solver and enables stress analyses of both real (e.g., imaged with SEM or OM) and simulated material microstructures. For one of its applications,  $\mu$ -FEA is being used to study stress concentrators, deformation, and microcracking around pores and

large grains and in secondary phases in materials subjected to a Hertzian contact stress gradient.

### Conclusions

Static and dynamic indentation and instrumented scratch testing facilities were established to quantify the loads and stresses associated with the initiation of quasi-plasticity and fracture processes. Numerous silicon nitride ceramics were investigated, and rate effects on hardness (i.e., quasi-plasticity) were studied. A new brittleness parameter was developed that enables static and dynamic indentation responses to be ranked for different ceramics. A



generic FEM was developed that can be used to predict the onsets of quasi-plasticity and fracture/cracking in any target material as a function of indentation load, indenter material, friction, and confinement. Lastly, microstructure-based finite-element analysis software ( $\mu$ -FEA) was copyrighted. The software enables stress analysis characterization at the microstructural scale and to help with the interpretation of quasi-plastic and fracture processes.

### **References**

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3. V. E. O. Bernhardt, *Z. Metallkde*, **33**, 135–144 (1941).
4. H. Li, A. Ghosh, Y. H. Han, and R. C. Bradt, *J. Mat. Res.*, **8**, 1028–1032 (1993).

### **Presentations and Publications**

A. A. Wereszczak and T. P. Kirkland, “Exclusivity of Strength-Limiting Extrinsic Surface and Hybrid Flaws,” in press, *Ceramic Engineering and Science Proceedings* (2006).

H. Wang and A. A. Wereszczak, “Mechanical Responses of Silicon Nitrides Under Dynamic Indentation,” *Ceramic Engineering and Science Proceedings*, **26**, 275–283 (2005).

G. Subhash, M. A. Marszalek, A. A. Wereszczak, and M. J. Lance, “Evaluation of Scratch Resistance and Residual Stresses in Machined Silicon Nitride,” *Ceramic Engineering and Science Proceedings*, **26**, 285–292 (2005).

H. Wang, A. A. Wereszczak, and M. K. Ferber, “Characterization of Dynamic Indentation of Silicon Nitrides by Using Vickers Indentation,” in preparation.

“Research Overview of ORNL’s Ceramic Science and Technology Group,” University of Alabama, Tuscaloosa, Alabama, April 20, 2005.

### **Special Recognitions and Awards/Patents Issued**

$\mu$ -FEA Software, M. K. Ferber and A. A. Wereszczak, U.S. Copyright Office, Certificate of Registration No. TXu1-234–195, 25 April 2005.

